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MICROSTRUCTURE ANALYSIS OF NA-NANODIAMOND PARTICLES

Dr. Tapan Chatterjee
Elias Jelis

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U.S. ARMY ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER

Munitions Engineering Technology Center

Picatinny Arsenal, New Jersey

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14. ABSTRACT

The purification process of detonation diamond nanoparticles was perfectly accomplished using nitric acid at high temperature and pressure. The transmission electron microscopy and electron diffraction technique revealed detonation diamond nanoparticles approximately 5 to 6 nm in diameter, similar to those obtained by distilled water purification. The energy dispersive analyzer from these perfectly well purified powdered materials showed a single carbon peak. Electron diffraction patterns confirmed a threefold symmetry, validating the elongated crystalline striations are aligned in a preferred 111 direction.

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INTRODUCTION

The nanodiamond sample was purified using a new technique of high temperature and pressure nitric acid, whereas the previous detonation diamond nanoparticle was washed with distilled water and purified by oxidation.

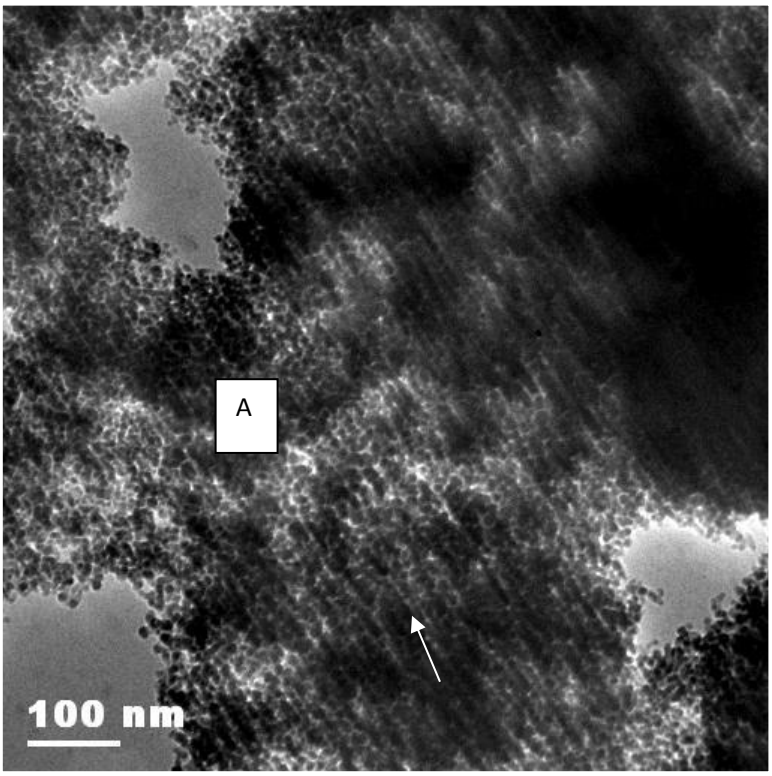
TRANSMISSION ELECTRONIC MICROSCOPY (TEM) ANALYSIS

Specimen Preparation

The 400 mesh coated grids were used for TEM analyses. Powdered samples were picked up by sharp pointed tweezers and placed on the coated grid. The specimen was also prepared by using methyl alcohol as a solvent. The Phillips 420 electron microscope at 120 KV voltage was used for TEM analyses.

Transmission Electronic Microscopy Results and Discussion

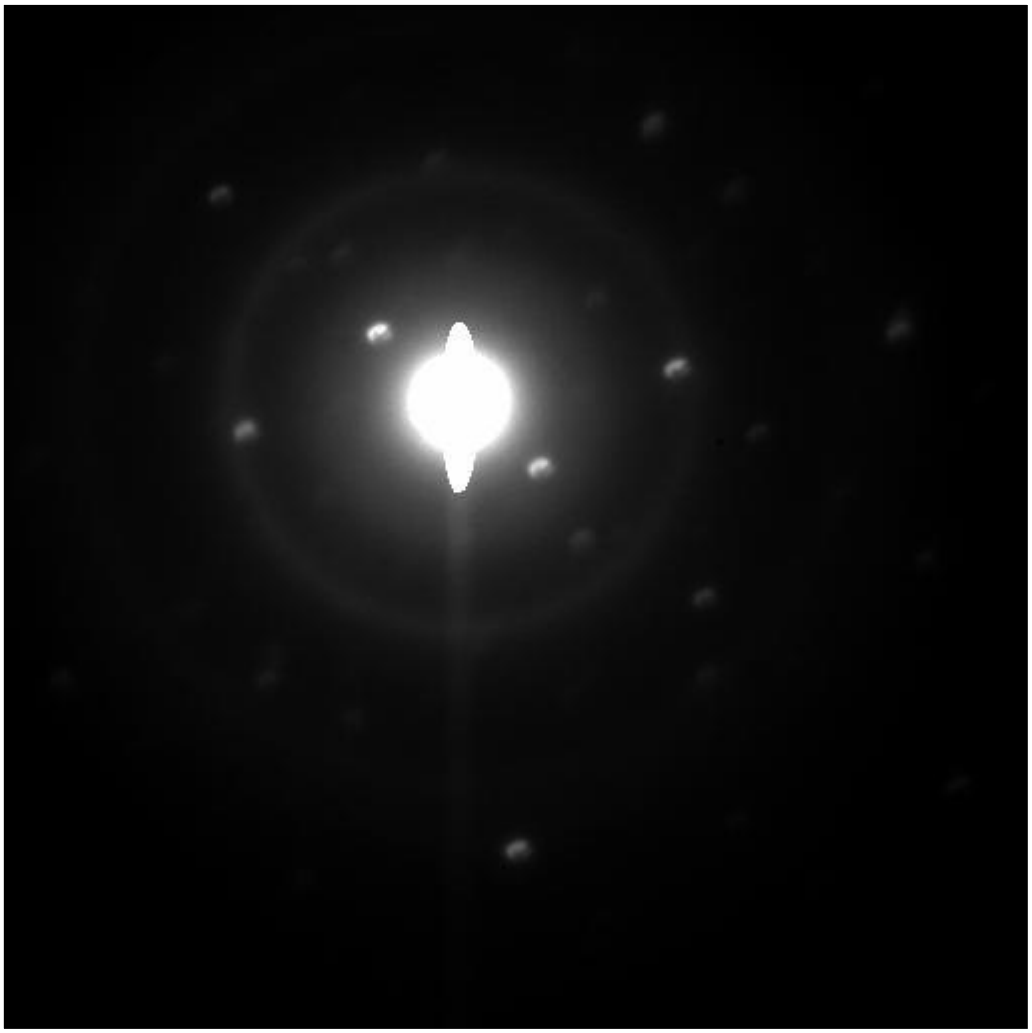
An electron micrograph obtained from a high-temperature and high-pressure purified nitric acid purified nano sample is shown in figure 1. The white arrow indicates one of many elongated striations composed of individual diamond nanoparticles aligned in a preferred orientation. The area A indicates arrays of a large number of such striations.



Note: The white arrow indicates a striation composed of individual diamond nanoparticles aligned in a preferred orientation. The area A indicates an array of a large number of such striations.

Figure 1
Transmission electron micrograph obtained from high-temperature and high-pressure purified nitric acid purified nano sample

A selected area electron diffraction pattern obtained from an area in figure 1 is shown in figure 2. This diffraction spot pattern indicates those elongated striations composed of nanoparticles are crystalline in nature and are aligned in a 111 direction. A large number of such diffraction spots observed on the TEM screen could not be captured digitally on the computer screen. A TEM picture obtained by a developer and fixing solution would provide a better selected area (electron) diffraction picture giving more information of the crystalline structure of these nanosamples.



Note: This diffraction spot pattern confirms the sample is crystalline and has a threefold symmetry confirming the elongated striations are aligned in a preferred 111 direction.

Figure 2
The selected area diffraction pattern obtained from an area in figure 1

Another electron micrograph from a different area of the same sample is shown in figure 3. This TEM micrograph reveals a large number of nanodiamond particles clustered together. The size of these nanoparticles is between 5 to 6 nm. Some of the micrograph area looks very dense black because of thick sample accumulation that the electron beam could not penetrate.

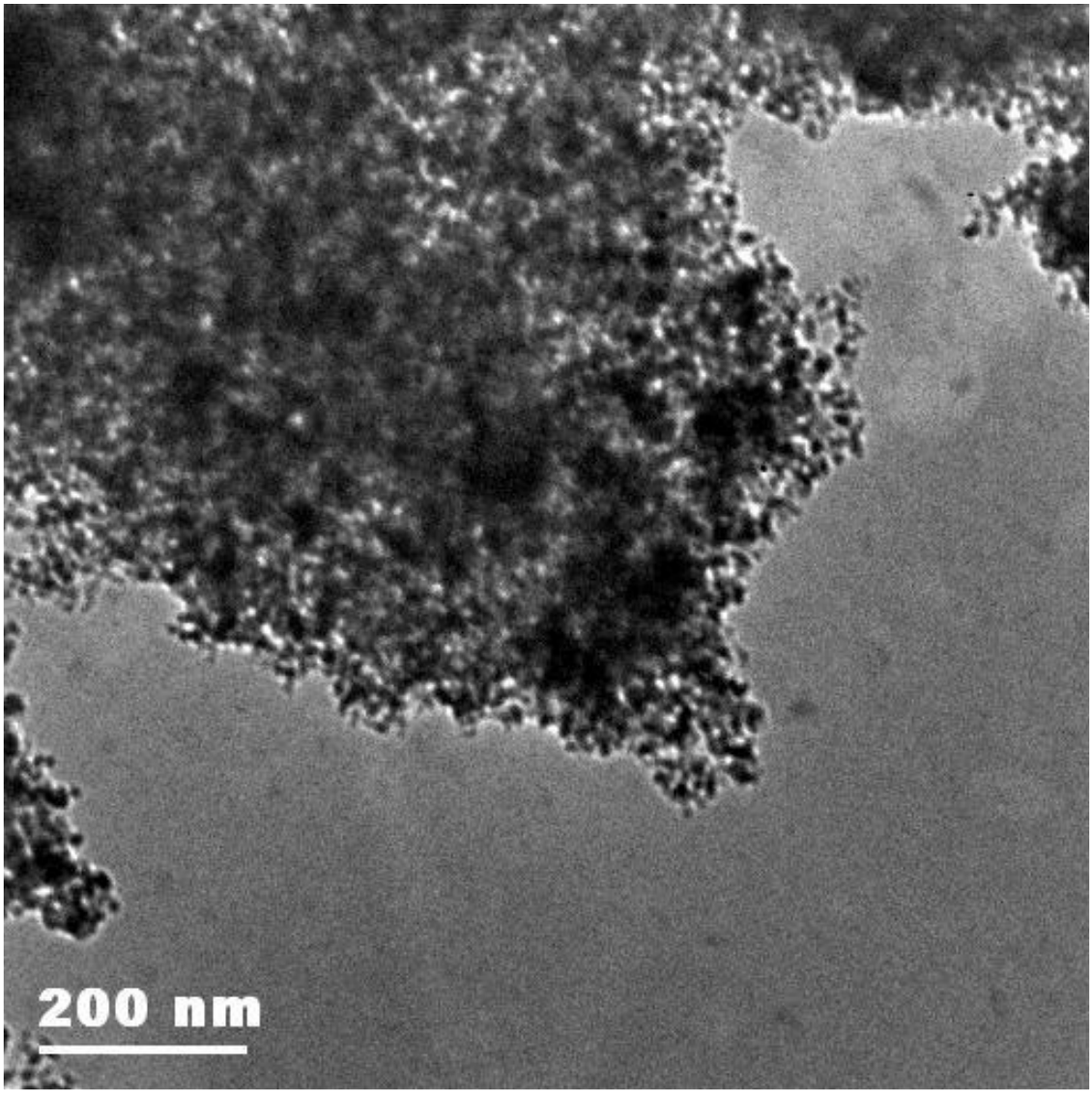
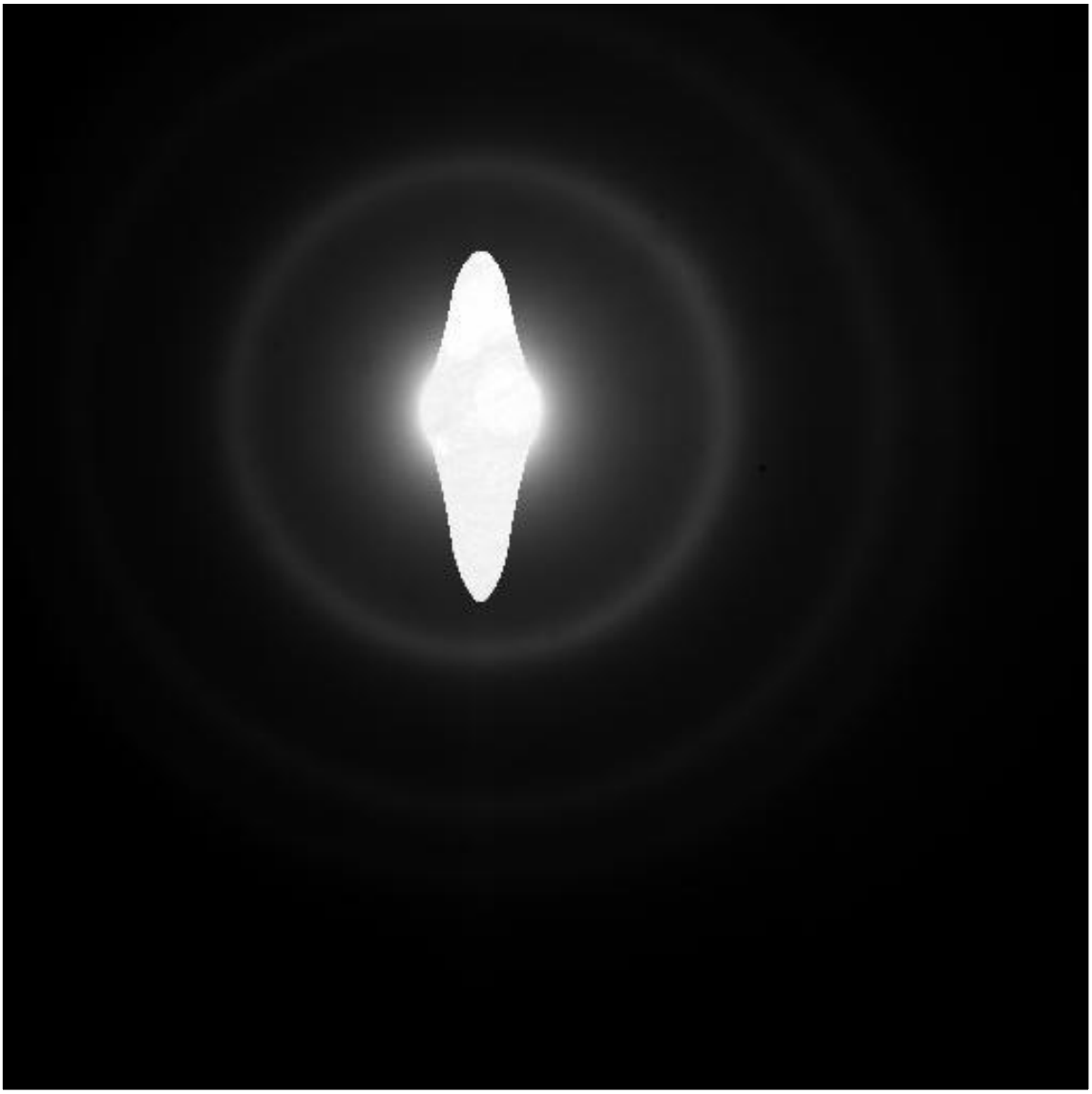


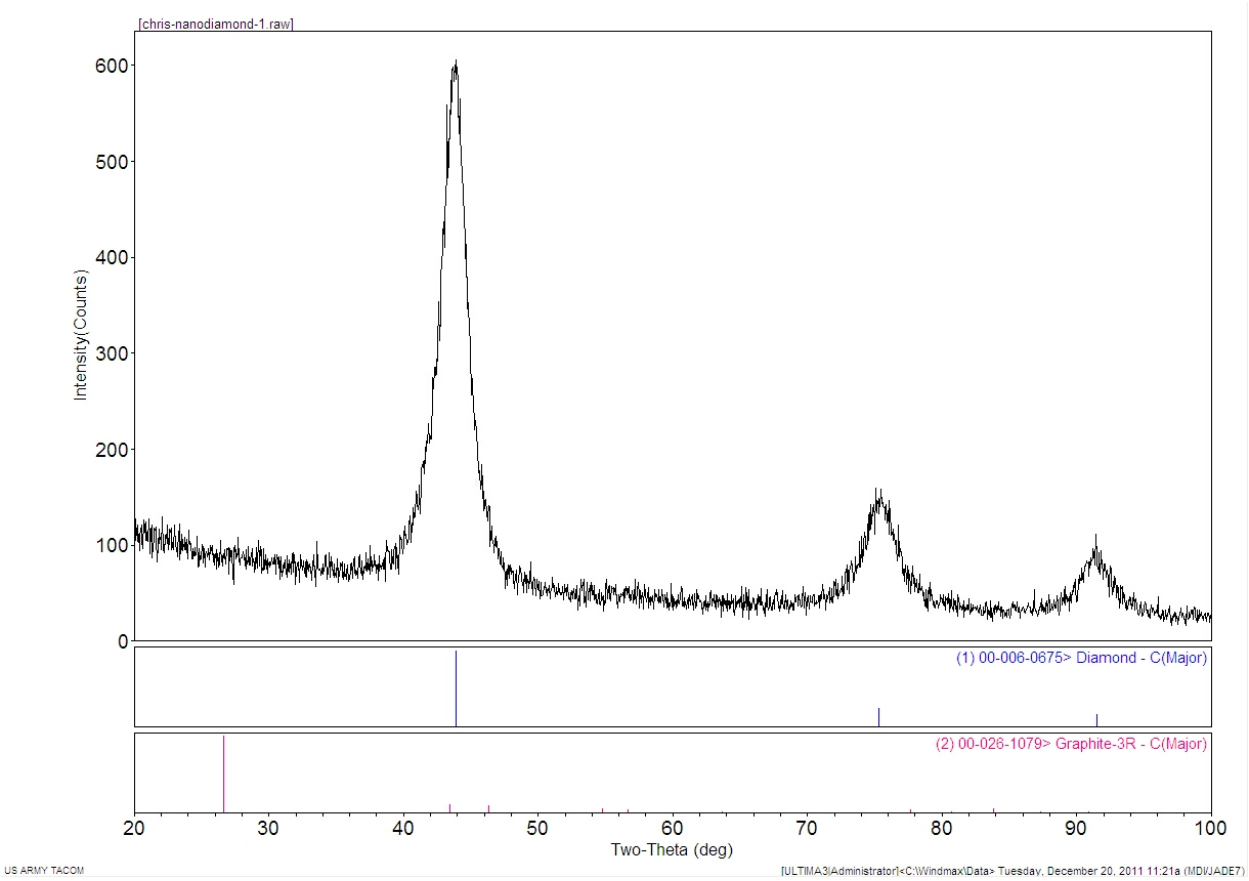
Figure 3
TEM picture taken from a different area of the 400 mesh grid does not show any elongated preferred striations; nanoparticles are randomly oriented

A selected area electron diffraction pattern from an area shown in figure 3 is shown in figure 4. This diffraction pattern reveals sharp faint circular rings hidden in broad diffraction rings. As it has been mentioned previously, these faint circular rings are visible on the TEM screen but could not be digitally reproduced on the computer screen. However, the circular diffraction rings that reveal the nanoparticles at this area of the sample are randomly oriented and crystallized as supported by the sharp peaks obtained by the x-ray diffraction (XRD) method in figure 5.



Note: Circular diffraction pattern confirms nanoparticles shown in figure 3 are randomly oriented.

Figure 4
Selected area electron diffraction pattern showing broad rings consisting of very faint hidden sharp rings



Note: The x-ray powder data files confirm the purified nanosample is diamond particles.

Figure 5
The XRD pattern from the powdered nan diamond sample shows sharp peaks confirming the sample is crystalline

SCANNING ELECTRON MICROSCOPE (SEM) ANALYSIS

Objective

Examine the morphology and elemental chemistry of detonated nanodiamonds (DND).

Experimental Procedure

The diamonds were simply spread onto an aluminum sample holder. Then, the sample was loaded into the Joel SEM and analyzed using energy dispersive spectroscopy (EDS).

Discussion of Results

Overall, the particle sizes range from about 25 μ down to the sub-micron range. More work using TEM will be completed to verify the sub-micron sized particles and to check for agglomeration. The composition of the particles contained 100% carbon; no other elements were detected (except for the specimen holder, which was made of aluminum).

Figures 6 through 8 show the SEM photographs of the DND particles taken using secondary electrons. The associated spectrum, figure 9, shows the composition of the particle analyzed in figure 3 (note the red X). The spectrum shows that the particle is pure carbon. There was some aluminum detected, but this was attributed to the aluminum sample holder. A trace amount of chlorine was detected, but this might be from handling the specimen holder (ie., sodium chloride).

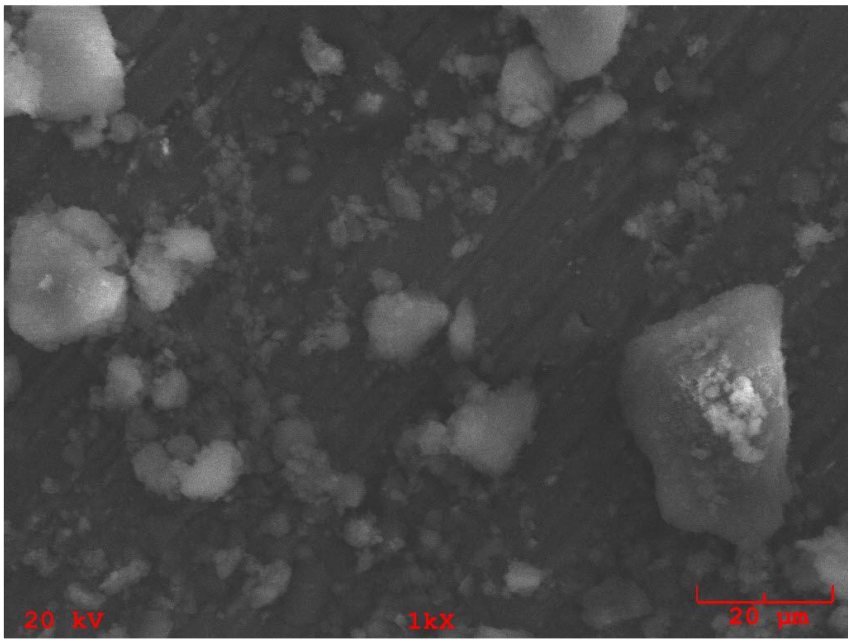


Figure 6
SEM photo of DND particles using secondary electrons

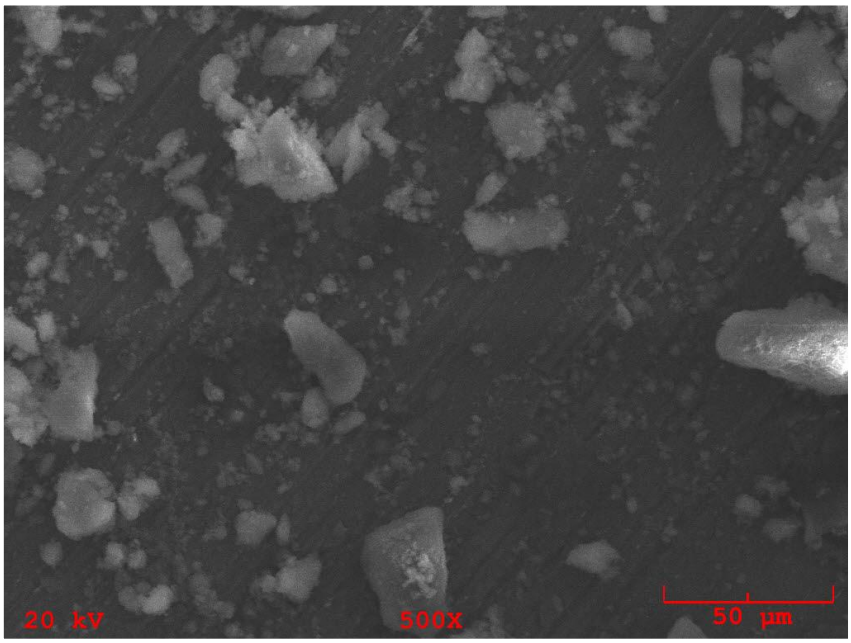
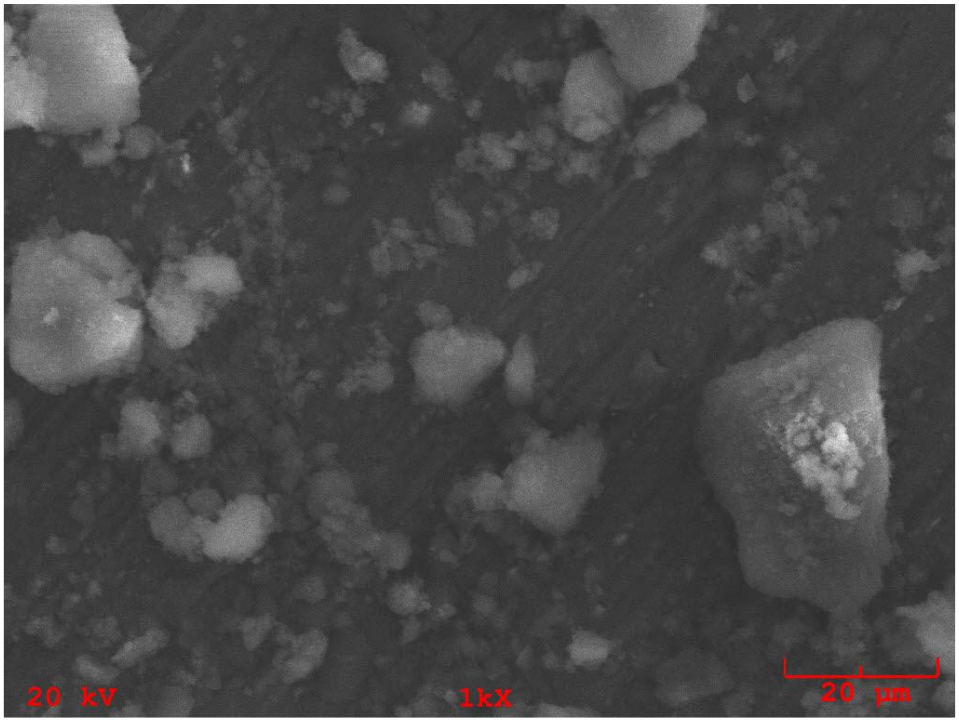
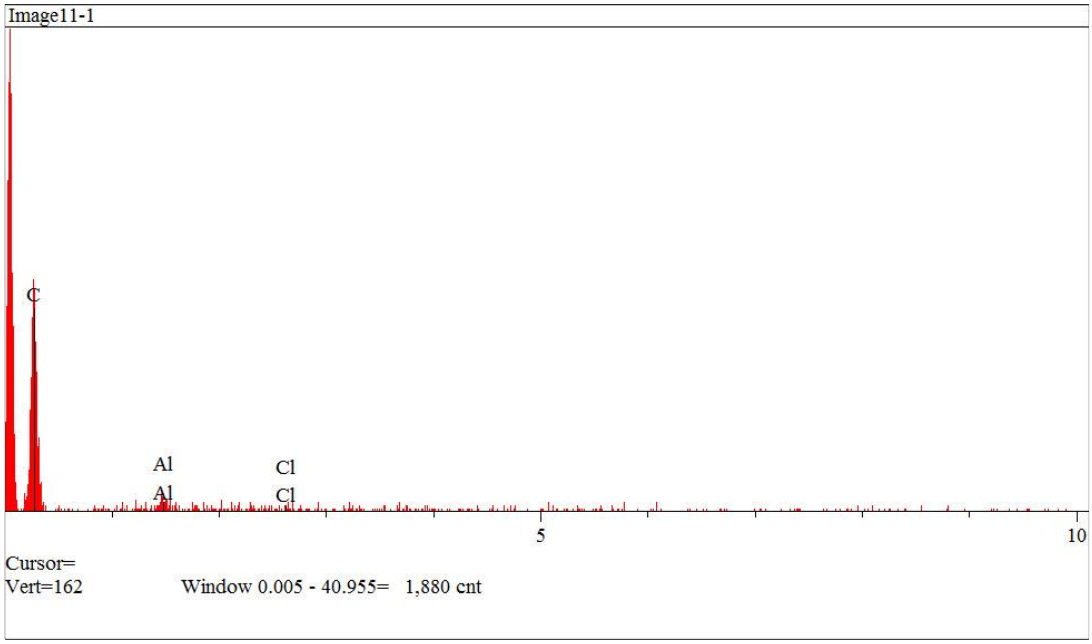


Figure 7
SEM photo of DND particles at a higher magnification (secondary electrons)



Note: The red X is the particle that was analyzed using EDS. The associated spectrum is shown in figure 4.

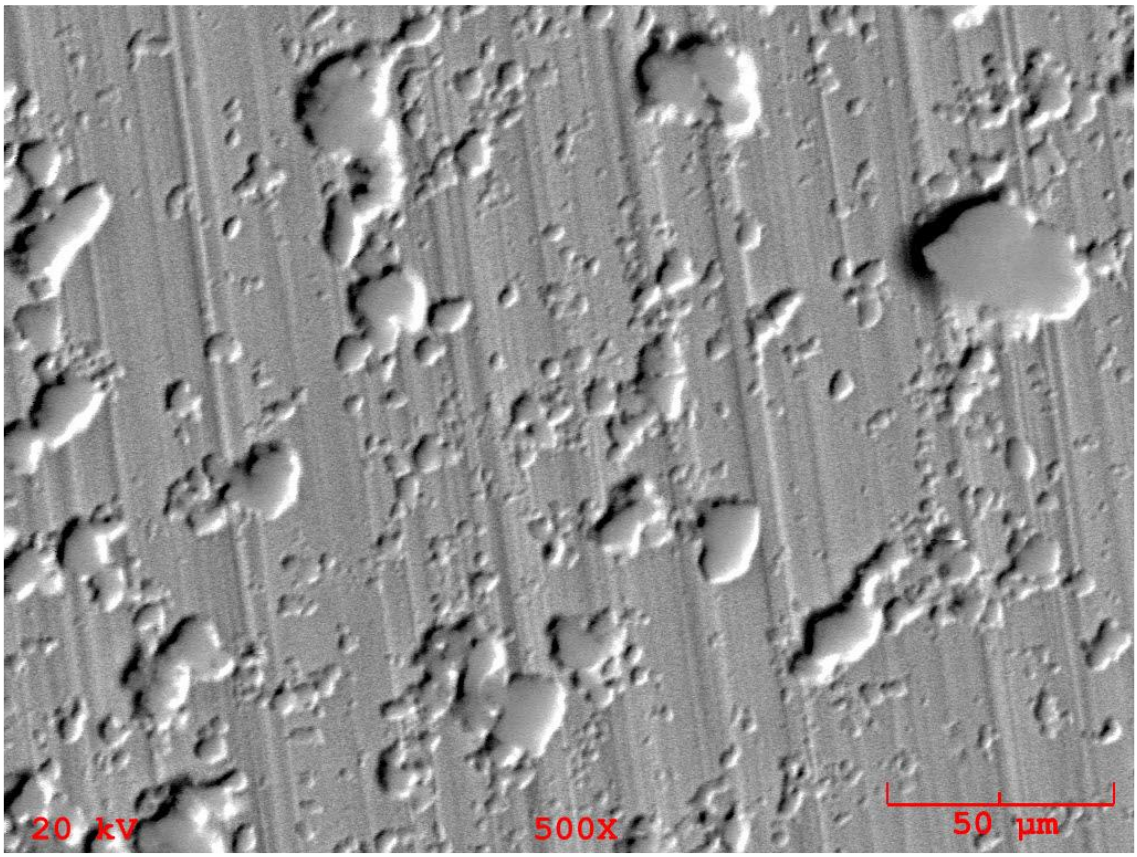
Figure 8
SEM photo of DND particles



Note: Carbon is the only element present. The aluminum is from the specimen holder and the trace amount of chlorine may be from handling the sample holder.

Figure 9
EDS spectrum of the particle in figure 3

Figure 10 is a SEM photograph of the DND particles taken using backscattered electrons – topography. In this mode, it is difficult to examine the structure of the DND particles, but it is easier to see the edges of the particles in order to measure the particle size. This photograph has been added for reference purposes to get an idea of the average particle size, but it does not account for agglomeration. Therefore, TEM needs to be done on this sample.



Note: The edges of the particles are more clearly defined, but they may be agglomerated.

Figure 10
SEM photo of the DND particles taken using backscattered electrons – topography

Point of Contact

The point of contact for this analysis is Stacey Kerwien, RDAR-MEE-M, Stacey.kerwien@us.army.mil.

CONCLUSIONS

Detonation diamond nanoparticles purified by high temperature and pressure nitric acid was perfectly well purified. The energy dispersive x-ray analyses showed a single carbon peak. The particle size of the pure diamond nanoparticles purified by this method is approximately 5 to 6 nm, same as those filtered by the distilled water and oxidation. The only difference between the two filtered processes is NA-nanodiamond particles are aligned in a preferred orientation in one area, and randomly oriented in other areas, and therefore not homogeneous. The other nanodiamond sample purified by distilled water and oxidation did not reveal this kind of microstructure.

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